Authors' response to Review 1

- Reviewer: Olga Yakubovich
- Citation: https://doi.org/10.5194/egusphere-2025-3879-RC1

Note: The reviewer comments are presented in **black bold print** in their original, unabridged wording. The author's response is displayed in blue.

The submitted manuscript is well written and presents a new methodological approach and algorithm for in situ (U–Th–Sm)/He dating of apatite aimed at reconstructing thermal histories of individual grains. Using apatite samples from southern Germany, the authors demonstrate that the proposed methodology enables the derivation of "He diffusion" profiles. It is shown that profiles corrected for parent nuclide distribution are flat in rapidly cooled grain, whereas in samples with heterogeneous parent nuclide distribution and complex thermochronological history, they exhibit non-flat geometries. It is shown how the obtained analytical data can be mathematically processed to derive information on the thermal history of the grains.

Thus, I consider this study to represent a valuable advancement in the development of in situ (U–Th–Sm)/He thermochronology.

We thank the reviewer for their time and careful review of our manuscript. The reviewer raises the following key concerns in their general comments below: Firstly, they state that for one of the apatites we analysed, we did not succeed in deriving a thermochronological path matching the observations in that grain. Secondly, they suggest that analysing well-characterised standard material would have been preferable to our choice of sample material. Lastly, the reviewer notes that some of our methodological approaches require a more detailed description. We address all comments below in detail and are optimistic that our answers and proposed revisions to the manuscript will alleviate the reviewer's concerns.

We have numbered the following reviewer comments (RC) and author responses (AR).

- **RC 1: My major concern is that the authors did not succeed in deriving a thermochronological path that adequately fits their observations for the BaF apatite. The obtained 4 profiles are reproducible and, as the authors noted, exhibit a counterintuitive geometry: the central part of the grain is significantly younger than the rims.**
- **AR 1:** We thank the reviewer for this comment. As they bring up several points at once, we will address each issue separately.
    - On the lack of success for deriving an adequate thermochronological path:
      Regarding the challenges in deriving an adequate thermochronological path for Apatite-BaF, we acknowledge that we were unable to invert all ^{4}He profiles of

Apatite-BaF for cooling histories. However, we want to emphasise that for profile Ap-BaF-P1, we did obtain fitting cooling histories (see Fig. 6 in our manuscript). We think a major issue with the non-invertible ^{4}He profiles is their asymmetry with respect to the c-axis, which conflicts with our inverse modelling approach that assumes approximately c-axis symmetric ^{4}He profiles. We will revise Section 4.7 in the discussion to highlight this limitation in our modelling approach. Furthermore, we suggest running forward models for Ap-BaF-P2 to Ap-BaF-P4 using the inversion results of Ap-BaF-P1 to assess whether the cooling history derived from Ap-BaF-P1 would fit the non-invertible profiles in Apatite-BaF.

- On the counterintuitive geometry of the ^{4}He profiles:
  For the ^{4}He profiles of Apatite-BaF, we observe that the ^{4}He concentration is highest at or near the grain centre, and the lowest at the grain rims. The profiles show a concave down geometry, as expected for a grain that has experienced diffusive loss of ^{4}He. The only exception to this pattern is Ap-BaF-P3, where the centremost spot exhibits the lowest ^{4}He concentration (Fig. 2 and Section 3.2, lines 232-237). We did not intend to suggest that the geometry of the ^{4}He profiles in Apatite-BaF (except for Ap-BaF-P3) is counterintuitive. We will clarify in Section 3.2 that, overall, the ^{4}He profiles for Apatite-BaF are rather inconspicuous.
- On the central part of the grain being younger than the grain rims:
  The pattern of younger in situ AHe dates in the grain centre of Apatite-BaF compared to the grain rim is counterintuitive and is most pronounced in Ap-BaF-P2 and Ap-BaF-P3. Ap-BaF-P1, which we successfully inverted for cooling histories, does not show this trend. Thus, the overall puzzling AHe date pattern did not preclude the derivation of potential time-temperature paths for Apatite-BaF. We will revise our discussion about the spatial variation of the in situ AHe dates in Apatite-BaF for clarification, also taking into account suggestions from RC 2.

- **RC 2: When the data contradict the model, it typically indicates either low-quality measurements (which I assume is not the case here) or that the model itself may not be correct. Thus, this issue should be discussed in detail. Why did the classical He-loss model fail to reproduce the results? The authors provide only a brief discussion of this matter. Among the factors considered are the presence of inclusions, uranium-enriched zones, and variations in the degree of crystallinity. However, as it was already mentioned, profiles are reproducible, no inclusions are observed in the analyzed half of the grain, and the effect of apatite crystallinity on helium diffusion is limited. The possible influence of implanted helium is not addressed.**
- **AR 2:** Thank you for highlighting the issue of helium implantation. As for RC 1, we will address each aspect separately below.
  - "Why did the classical He-loss model fail to reproduce the results?"
    We are not entirely sure what this question alludes to. Assuming the comment targets the counterintuitive in situ AHe date pattern, for which we admittedly do not have an explanation, we suggest further expanding the discussion and elaborating on the variability in crystal lattice properties and diffusivity not directly related to the parent nuclides but to variations in major element composition (e.g. Djimbi et al. 2015) or vacancy damage (e.g. Gerin et al. 2017), which we have neglected to mention.

However, with the information we have for Apatite-BaF, we cannot determine whether these factors are significant.

- "The possible influence of implanted helium is not addressed."

  We thank the reviewer for pointing this out and recognise that a discussion of this topic should be added to the manuscript. We do not think that ^{4}He implantation has a significant influence on the ^{4}He profiles in Apatite-BaF for the following reasons:
  1. When evaluating the potential impact of implanted ^{4}He from external sources, it is commonly assumed that the outer 20 µm rim of the apatite grain is affected the most (e.g. Spiegel et al., 2009; Gautheron et al., 2012). For our measurements, the distances of the in situ ^{4}He ablation spots to the grain rims are larger than 20 µm, except for two in situ ^{4}He ablation spots that were neither included in thermal modelling nor (U-Th-Sm)/He age calculations (refer to Fig. 2 and Table 3 in the manuscript for spot distances to the grain rim).
  2. Furthermore, models of implantation scenarios predict a significant peak in ^{4}He concentration at the grain rim facing the external ^{4}He source (see, for example, Fig. 6 in Gautheron et al. 2012). However, our Apatite-BaF ^{4}He profiles do not show this peak, suggesting that the impact of ^{4}He implantation is not significant.
  We will add a paraphrased version of the above elaboration to the discussion section of our manuscript for clarification.

- **RC 3: This point is of critical importance—if the model does not accurately describe observed in situ (U-Th-Sm)/He profiles, its validity as a basis for thermal history modeling becomes questionable.**
- **AR 3:** In our opinion, the modelling results for both Apatite-URG and Apatite-BaF clearly demonstrate that in situ ^{4}He profiles can be inverted for plausible thermal histories (Fig. 5 and Fig. 6 in our manuscript). Given that zoned or heterogeneous grains such as Apatite-BaF are known to be problematic for conventional whole-grain (U-Th-Sm)/He approaches where rigorous criteria for optimal grain selection apply, we do not see the rationale in dismissing the in-situ ^{4}He profile approach based on difficulties with interpreting the heterogeneous Apatite-BaF.
  In our opinion, the validity of the in situ approach hinges on the ability to produce accurate results for homogeneous grains (as for whole-grain (U-Th-Sm)/He dating). We want to emphasise here that the homogeneous Apatite-URG yielded good results, and we thus consider the method valid. Apatite-BaF, on the other hand, highlights the limitations and areas of further development.
  We suggest modifying our manuscript in several places to 1. explain our choice in analysing homogeneous and heterogeneous grains (testing the validity and limitations of the approach), 2. explicitly clarify that homogeneous grains are the most suitable and straightforward choice for routine analyses and 3. state that zoned or heterogeneous grains, such as Apatite-BaF, can be analysed, but are not recommended.

General comments

- **RC 4: For a study of this kind, it would have been preferable to use apatite grains from thermochronologically well-characterized localities, where FT and AHe ages are already available, or where even 4He/3He profiles have been**

obtained. While this may not be essential from a methodological point of view, it would undoubtedly strengthen the testing and validation of the proposed thermochronological modeling approach.

- **AR 4:** This is a valid criticism, and we agree with the reviewer that a well-characterised sample or standard material would have allowed for better comparability of our approach to whole-grain (U-Th-Sm)/He dating and 4He/3He analyses.
  Unfortunately, the well-characterised standard material available in our laboratory (Fish Canyon Tuff apatite (FCT) and Durango apatite) was not suitable for our measurement approach. For Fish Canyon Tuff, the available grains were too small. Further, zonation has been identified as a concern for this standard (e.g. Pickering et al. 2020). For Durango apatite, the grains were too large for the analysis to be performed within a reasonable cost and timeframe.
  For this reason, we chose Apatite-URG (from a Miocene tuff with an independently constrained U-Pb age) as a substitute for standard material. The Apatite-URG sample provided large, euhedral and clear crystals. Furthermore, the expected cooling history for Apatite-URG is simple and sufficiently well-constrained due to the volcanic nature of the source rock and the independently determined U-Pb age. Therefore, we consider this sample to be appropriate for testing and validating our methodology.
  We will add a few sentences explaining the choice of sample material to our manuscript.

- **RC 5: Sample preparation involved SEM imaging and mounting in Teflon, both of which exposed the samples to elevated temperatures. The authors likely used low current values for SEM imaging and low temperatures for mounting to limit possible thermal effects; thus, it should not affect the AHe ages. But detailed information regarding this issue should be presented in the methodological section.**
- **AR 5:** Thank you for pointing out the lack of discussion concerning the thermal effects of sample preparation. We will add the Teflon embedding temperature and duration (300°C for 2 minutes) as well as the SEM imaging settings (beam current 15 kV) in the methods section.
  - Concerning the potential effect of Teflon embedding, we can calculate the fractional He-loss resulting from such a short-term heating event using the approximation from Reiners et al. (2007) (Equation 3). With the diffusion parameters determined for the Durango apatite standard (Farley, 2000) as an estimate and using our measured grain radii, we can approximate that the fractional He-loss from Teflon embedding is ~1% for grains the size of Apatite-BaF and ~0.5% for grains the size of Apatite-URG. We will add this information to the methods section.
  - The impact of SEM analyses on the AHe system has been systematically examined by Shan et al. (2013). They found that AHe dates are not significantly affected by SEM. They also determined that the majority of heating caused by SEM would be limited to the upper 0.3 µm of the grain. We will refer to Shan et al. (2013) in the methods section.

- **RC 6: What are the reasons for the significant variations in He extraction pit depth observed within a single grain (for example, from 6.9 to 9.3 µm; approximately 25%; Table 3)? If the laser energy is dissipated/reflected, could it**

potentially heat the sample (mobilize He) or heat internal parts of the cell (increase blank level)?

- **AR 6:** We report the maximum pit depth in Table 3. Due to the unevenness of the pit floor, the maximum pit depth can vary significantly. However, the variation in maximum pit depth does not necessarily mean the ablated volume strongly varies (Figure 1). For example, in Apatite-URG, the maximum pit depth ranges from 6.9 to 9.3 µm, but the standard deviation of the mean pit volumes is only 4%. We suggest removing the maximum pit depth information from the manuscript and only reporting the mean pit depth.
  We are unsure what the second comment regarding the dissipation of laser energy, the mobilisation of He from heating of the sample and the increase in blank level allude to in the context of pit dimension variations. As for the heating of the grain outside the pit during He extraction, Boyce et al. (2006) reported that laser heating and He degassing outside the ablation pit are negligible.

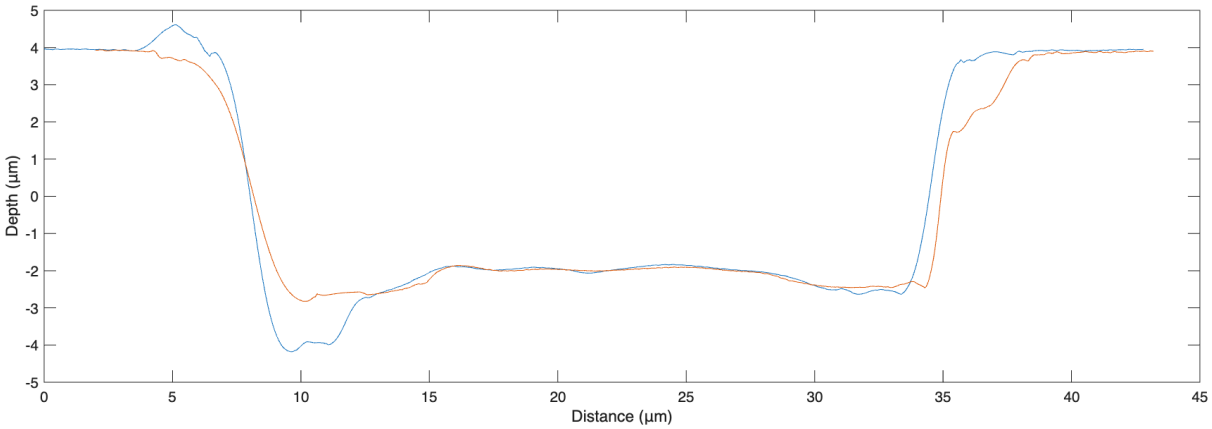

Figure 1: Cross-section through two exemplary ablation pits with significant variation in max. pit depth, but quite consistent pit volumes.

Additional minor comments can be found within the attached file.

Comments from the attached pdf file

Note: The minor comments have been marked and annotated in the text and figures. We have copied the reviewer's comments and added the line information. For reference, we have also quoted text fragments marked by the reviewer and indicated which section of the manuscript the reviewer's comment concerns.

Abstract

- Line 21 ("for our study and instrument set-up"): **provide extra details (Resochron, eximer etc)**

- We will add “RESOchron system (Applied Spectra), consisting of a He-line and an excimer laser”.

- Line 24 (“20-30microns”): **you tested only 20 microns, aren't you?**
  - In this sentence, we meant to make a recommendation for an optimal spot diameter. We will rephrase this sentence and add another sentence detailing the range of spot sizes tested (10 – 30 µm) and the respective grains’ AHe dates.

- Line 24 (“six-spot in situ 4He profile requires a minimum grain diameter (measured perpendicular to the c-axis) of 145 µm”): **is it true for all apatites or only for the young ones?**
  - This is our general recommendation. With an optimal ablation spot diameter of 20 µm, a grain size of at least 145 µm is needed to measure a ^{4}He profile composed of six individual in situ ^{4}He measurements. (Details are in section 4.1 of our manuscript.) We will modify this sentence to clarify this, e.g. by writing “ Additionally, **with a ^{4}He ablation spot diameter of 20 microns**, a six-spot in situ ^{4}He profile requires a minimum grain diameter (measured perpendicular to the c-axis) of 145 µm”.

- Line 26 (“potentially including zoned and irregularly shaped crystals”): **so you suggest that BaF -like apatite can be used for modeling?**
  - Yes, we suggest that heterogeneous “BaF-like” apatites can be used for modelling.

Methods

- Line 85 (“age”): **U-Pb**
  - Thank you for pointing this out. We will add U-Pb before “age standard” for clarification in line 85.

- Line 90 (“embedded in a Teflon sheet”): **At what temperature?**
  - The hotplate used for embedding the grains in Teflon was set to 300°C, and the mount remained on the hotplate for 2 minutes. We will add this information.

- Line 92 (“SEM”): **SEM heat the grains. What energies have you used? What was the beam current? Were the samples coated? By gold or by coal?**
  - We did not coat the sample mount. For the SEM scan, we used a voltage of 15 kV. The emission current was 40.3 µA, the filament power was 4.46 W, and the dwell time was 200ns. We will add this information to the manuscript.

- Table 1 **Which coordinate system? WGS84 or ETRS89?**
  - We use WGS84. We will add this information to the table notes.

- Line 108 (“Line blanks were recorded regularly”): **It is cold blank. What about the hot blank? Have you measured the blank when ablate helium free sample?**

- Each sample mount is individually placed in an ultra-high-vacuum (UHV) laser cell and evacuated for a minimum of 24 hours to ensure that vacuum levels reach the low-pressure regime required for high-precision He analysis. In fact, our cold cathode was unable to measure the pressure since it reached values below the detection limit 1E-8 torr. Before analysing samples, several initial blank analyses are run until blank 4He/3He ratios reached values <0.0001. These blanks use the identical valve and volume configuration as the subsequent sample measurement, but without firing the laser.

- Line 109 ("0.0003 to 0.0005 ncc"): **How do you calibrate the MS?**
  - Quantification of radiogenic ^{4}He extracted from individual laser pits relies on calibration against an internally monitored 4He reference reservoir ("Q-tank"). The ^{4}He volume of the Q-tank is known but not constant: it undergoes predictable exponential depletion as Q-shots are consumed during routine analytical work. To maintain accuracy, the Q-tank is recalibrated at regular intervals, typically annually or after several thousand Q-shots, using a secondary reference reservoir ("D-tank"). The D-tank contains an independently certified amount of ^{4}He, externally calibrated by Des Patterson, and serves as the long-term stability anchor for all Q-tank recalibrations. Calibration factors derived from these intercomparisons are used to convert measured ion currents into absolute ^{4}He amounts in sample analyses.

- Line 111 ("after blank correction"): **have you incorporated the blank uncertainty into standard deviation calculations?**
  - Yes, blank uncertainty is propagated into the ^{4}He concentration standard deviation. We will report the equations in the supplementary material.

- Line 122 ("re-polished grain surfaces"): **You have removed 10 microns or more?**
  - Thank you for this question. From the pit depths, we can estimate that ~10 µm had to be polished away to smooth the grain surface. We were careful in repolishing the mount and checked the state of removal multiple times during the process to make sure we stopped polishing as soon as the pit traces had been removed. We will expand the methods section and provide more details about the repolishing procedure, e.g. by adding **"Prior to parent nuclide measurements, the grains were polished for 3.5 h on a polishing machine at intervals of 4 to 20 min with a decreasing force from 20 to10 N to remove the helium extraction pits and create an even surface for U, Th and Sm distribution mapping. The state of removal was checked multiple times during the process, and polishing was stopped as soon as all visible pit traces had been removed."**

- Line 124 ("Durango"): **Durango?**
  - We will add "age standard for apatite" and the reported standard (U-Th)/He age in parentheses for clarification.

- Table 2 ("24"): **You have a large variations in pit depths for He measurements, and quite a constant values for trace elements. Why?**

- Thank you for this question. We observe variations in the depths of the ^{4}He ablation pits, but not in the depths of trace element pits, as the former are individually measured, while the latter is a calculated value. We measured the pit dimensions of each ^{4}He ablation pit individually because the pit volume is necessary for ^{4}He concentration calculation (lines 111 – 114 in the manuscript). The pit depth for the trace element measurements, on the other hand, is a calculated value that we derived from the established ablation-time-depth relationship (lines 129 – 130 in the manuscript). For the trace element measurements, our interest only lies in the depth in the grain from which the signal originates, not in the exact pit dimension. Hence, the calculated value suffices. We will clarify this in the table notes. We will also replace "Mean spot depth" with "Mean pit depth" in the header of Table 2 (and where relevant throughout the manuscript) and add the ^{4}He pit depth SD for clarity.

3. Results

- Line 213 ("of 2.8E15 ± 5.0E15 at g-1 to 8.5E15 ± 2.5E15 at g-1")**: why haven't you used 20 microns spot for McClure? It would allow you to receive a profile, wouldn't it?**
  - The reviewer raises a good point. We wanted to test different ^{4}He ablation spot sizes to test the limitations of the in situ ^{4}He measurements concerning spot size, and thus used a different ablation spot diameter for each analysed grain. We anticipated a higher ^{4}He concentration in Apatite-McClure and therefore chose to test our smallest spot diameter in this grain. We fully agree with the reviewer that, in hindsight, a bigger spot would have been the better choice.

- Line 250 ("Apatite-McClure displays minor internal variation (…)"): **McClure apatite is well-described. Given that this data is not used in modelling I would suggest it be redundant**
  - While Apatite-McClure highlights the limitations of our method, especially concerning ablation spot size, we agree that it is of minor importance compared to Apatite-URG and Apatite-BaF. We suggest removing detailed descriptions concerning Apatite-McClure and moving the remaining information that is relevant to the manuscript to the appendices.

- Line 253 ("slight (…) 5-17 ppm"): **slight? 3 times difference**
  - Both the U-concentration and the variation (5-17 ppm) are small compared to Th (86–234 ppm) and Sm (20–310 ppm). We will rephrase this sentence to clarify that the total amount and variation in U is small or "slight" relative to Th and Sm.

- Line 283 ("83.22 ± 9.55 Ma to 162.25 ± 28.95 Ma"): **round the values appropriately**
  - Thank you for pointing this out, we will do that throughout the manuscript.

- Line 293 ("was 20 Ma"): **why not 16.75, or 16.75+0.84?**
  - We concede that the model box is larger than strictly necessary. We wanted to avoid the starting point dictating the inversion result. Given that the U-Pb and (U-Th-Sm)/He dates for Apatite-URG overlap, we drew a larger exploration box, including

up to 20 Ma. Ultimately, this was larger than necessary, but as it does not influence the AHe date, it is a question of presentation.

- Line 305("mean apatite U-Pb date derived from trace element measurements in Apatite-BaF"): **add the U-Pb data as a supplementary file**
  - The U-Pb data are already provided in the data supplement linked on Zenodo. We will refer to the supplement with a link in the text.

- Line 306 ("near the sample location"): **where exactly? 1 km - 10 km distance? same geological unit?**
  - Thank you for this comment. Unfortunately, we can only estimate the distance of our Apatite-BaF sample location to the sampling sites of Vamvaka et al. (2014) since they do not provide coordinates, but only an overview map.
    We estimate that the distance between our Apatite-BaF sample location and the nearest sampling site of Vamvaka et al. (2014) is approximately 5-10 km. For this nearest sample, they report that the sampled rock type is granite, which aligns with the lithology of our sample. However, we cannot be certain that we have sampled the same intrusion.
    Vamvaka et al. (2014) suggest that an exhumation-and-reheating scenario is generally plausible for the Bavarian Forest. Our intention with the comparison of our Apatite-BaF to findings from Vamvaka et al. (2014) was to assess the plausibility of the derived cooling history for Apatite-BaF, not to prove its accuracy. We will emphasise this in Section 3.5.1.

- Fig.5 and Fig. 6: **what are those artefacts near the center?**
  - Thank you for this important question. We will expand Section 2.5 to clarify the reasons for the artefacts, and we will also add a brief explanation to the figure captions of Figs 5, 6, and 7.
    The forward-modelled ^{4}He profiles display a "jump" near the centre, because they merge two core-rim profiles. In theory, for a perfectly homogeneous grain, only one core-rim profile would be needed, since both halves of the grain (from core to rim) would yield identical information. The forward model we used (detailed in Glotzbach & Ehlers, 2024) is designed for displaying such core-rim profiles.
    To model a full rim-rim profile, which is vital for asymmetric ^{4}He profiles (heterogeneous grains), we combine two core-rim profiles. Since both core-rim profiles share a common point at the grain centre, this centre point would be defined twice in the model. Therefore, it must be excluded (left undefined) in one of the profiles. As a result, the concentration appears to "jump" where the profiles meet.

- Line 332 as well as one apatite grain from the McClure Mountain Syenite standard (Apatite-McClure): **no 4He profiles for McClure**
  - We will remove information about Apatite-McClure from this section.

- Line 335 ("The measurement uncertainties for Apatite-McClure (ablation spot diameter 10 µm) exceed 40%"): **it is not a main result, i guess**
  - We will remove the information about Apatite-McClure from this section.

- Line 346 (“sensitivity testing”): **new paragraph**
  - We will add a new paragraph.

- Line 349 (“the high individual measurement uncertainties for 4He”): **The used ablation pit was too small. Nothing wrong with McClure - 4He concentrations are high enough. Please, rephrase**
  - We will remove the sentences referring the Apatite-McClure from this section.

- Line 355 (“and heterogeneous grains”): **part of heterogeneous?**
  - We are not entirely sure what the comment intends to point out here. We suggest rephrasing the sentence for better clarity, e.g. “In situ ^{4}He profiles can be inverted for cooling histories of homogeneous and, even though more challenging, heterogeneous grains.”

Discussion

- Line 376 (“SD<5%”): **see table 3, SD is > 5%, while 3 blanks level is achieved**
  - We thank the reviewer for this comment. The sentence is poorly worded, and we will rephrase it.
    We ideally want a signal clearly above 3x the blank level with a standard deviation (SD) <5%. For our instrument set-up, measurements that are only slightly above 3x the blank level typically have blank-corrected SDs around 6-7%. Generally, the lower the difference between the signal and the blank, the higher the uncertainty in the measurements.

- Line 423 (“older dates towards the grain rim and younger dates towards the grain centre”): **It is a bit misleading, as URG might not have this trend (your next sentence)**
  - We will rephrase this section and shorten it by removing most references to Apatite-URG. We will only discuss Apatite-BaF in detail.

- Line 429 (“In both cases, the observed date distribution within the grains is counterintuitive”): **You don't have data to proof this trend for URG sample.**
  - We will remove sentences discussing Apatite-URG.

- Line 437 (“From our data, we cannot decipher the reason for the observed inverted in situ date pattern”): **this should be discussed in the detail! what is the most likely scenario? what about alpha implantation? whats is an average U and Th content of a host rock?**
  - We will add elaboration on the impact of ^{4}He implantation as per our detailed response to RC 2.

- Fig. 7 **why near the zero value do you have this mathematical artefact**

- Please refer to the above explanation for Figs. 5 and 6.

- Line 510 ("accurately"): **the error is quite large**
  - We agree with the reviewer's point that the measurement uncertainty is relatively large. Although it may be a semantic question, it is crucial to distinguish that larger measurement uncertainties do not imply inaccuracy, but rather imprecision. The thermal histories we derive from the ^{4}He profiles for Apatite-URG all show very rapid cooling in the Miocene, which aligns with expectations for a volcanic rock, such as the source rock of Apatite-URG. Furthermore, the timing of cooling is consistent with the independently determined U-Pb age. Thus, we maintain that the derived cooling history is accurate, even though the ^{4}He profile measurements are arguably imprecise.

Conclusion

- Line 558: delete "Using"
  - Ok, we can delete the word.

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